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AUSTENITIC GRAIN GROWTH CHARACTERISTICS IN RAPIDLY SOLIDIFIED MARTENSITIC STEELS

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ABSTRACT

Comparison of the austenitic grain growth characteristics of conventionally processed and rapidly solidified 9Ni-4Co and 2Mo steels has revealed an unusual resistance to grain coarsening of the rapidly solidified material. At 1200°C where the conventionally processed material coarsens to a grain size of several hundred microns, the rapidly solidified material retains a grain size of $\sim$20 μm. The morphological characteristics of the inhibited grain growth indicate strong pinning at prior powder particle boundaries. Long-time austenitizing treatments at 1100-1200°C eventually lead to discontinuous coarsening. Evidence so far suggests that the unusual coarsening resistance may be due to finely distributed sulfides. It is anticipated that high austenitizing treatment of rapidly solidified steels will allow improvement of sharp-crack fracture toughness (K_{IC}) without the loss of Charpy energy and tensile ductility associated with excessive grain coarsening; exploitation of this phenomenon, however, will require an extreme level of cleanliness in powder processing.

Introduction

This study is a continuation of work presented at the previous rapid solidification processing conference in 1977 (1) as part of an ongoing program on the physical metallurgy of rapidly solidified steels with the ultimate objective of improved mechanical properties. In the previous study, the microstructures of a secondary hardening martensitic Ni-Co steel (HP 9-4-20) were compared after conventional and rapid solidification processing (RSP). Other than a more uniform distribution of alloy carbides and slightly finer grain size, the rapidly solidified material did not reveal a striking difference relative to the conventionally processed material when given a conventional heat treatment. In the present study, austenitizing treatments were extended to very high temperatures revealing a dramatic difference between the grain coarsening resistance of rapidly solidified and conventionally processed materials.

Materials and Experimental Procedures

Compositions of the steels investigated are given (in wt. pct.) in Table I. These include four 9Ni-4Co steels with carbon levels from 0.2% to 0.8%, and a simple 2Mo secondary-hardening steel. Two vacuum-induction melted ingots of each composition were prepared. One ingot of each pair was converted to rapidly solidified powder by the Pratt and Whitney centrifugal atomization process and the resulting -140 mesh powder consolidated by hot extrusion under the conditions given in Table I. For comparison purposes, the companion ingots were processed conventionally, involving hot forging and hot rolling to 0.5 in. plate in the case of the 9Ni-4Co steels and 1-1/8 in. bar in the case of the 2Mo steel.

TABLE I

STEEL COMPOSITIONS

	C	Mn	Si	P	S	Ni	Co	Cr	Mo	V	Al
RSR 75	0.22	0.51	0.09	<0.010	0.004	8.95	4.45	0.75	0.98	0.09	0.026
RSR 78	0.41	0.24	0.10	<0.010	0.005	7.8	3.95	0.28	0.27	0.09	0.037
RSR 81	0.63	0.27	0.09	<0.010	0.006	7.7	3.95	0.27	0.27	0.09	0.032
RSR 82	0.83	0.28	0.09	<0.010	0.007	7.7	3.9	0.28	0.28	0.09	0.032
RSR 187	0.26	1.05	0.05	0.005	0.007	--	--	--	2.04	--	0.005

Extrusion Conditions

RSR 75	820°C, 12:1
RSR 78	840°C, 10:1
RSR 81	840°C, 10:1
RSR 82	840°C, 10:1
RSR 187	840°C, 9.5:1

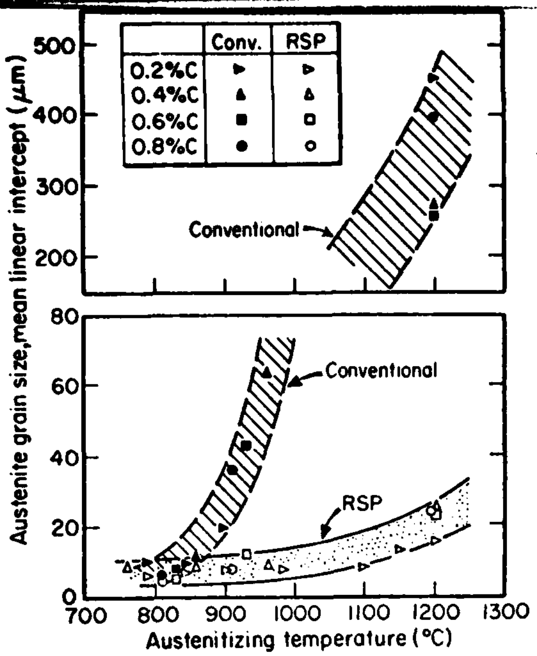

Fig. 1. Austenite grain size of 9Ni-4Co steels vs. temperature for one hour treatments

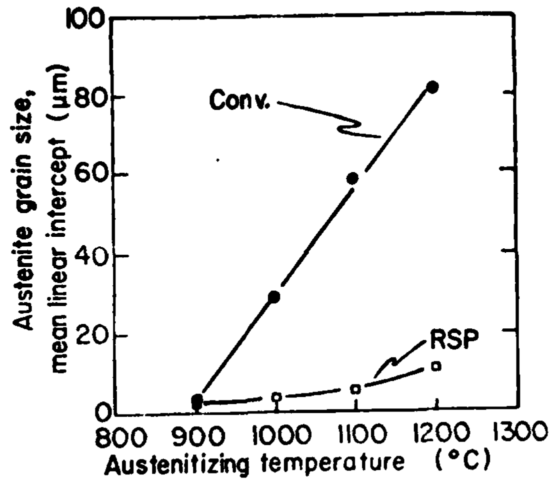

Fig. 2. Austenite grain size of 2Mo steel vs. temperature for 15 minute treatments.

Specimens for grain-size measurements were sealed in quartz capsules, back-filled with high-purity argon, and were water quenched after heat treatment. To reveal the prior-austenite grain boundaries via optical metallography, the specimens were given a subsequent temper embrittlement treatment at 500°C for three days. Longitudinally sectioned metallographic specimens were polished and etched in a solution of 1 drop HCl in 100 cc saturated picric acid with a wetting agent at 65°C for 20 minutes. The black film was removed by a light final polish. Grain sizes were measured by the mean linear-intercept method, counting 400 intercepts on average.

Results and Discussion

The austenite grain size vs. temperature for one-hour austenitizing treatments is shown for the 9Ni-4Co steels in Fig. 1. The conventionally processed material maintains a fine grain size below 900°C as is typical for an Al-killed steel with 0.1 V (2). Above 900°C, rapid grain coarsening ensues. In contrast, the RSP material resists grain coarsening to temperatures as high as 1200°C, maintaining a ∿20 μm grain size where the conventional material has coarsened to several hundred microns. The grain-growth behavior was not significantly influenced by carbon content.

Similar results for the 2Mo steel after 15 min. austenitizing treatments are shown in Fig. 2. Again the conventional material coarsens rapidly above 900°C, while the RSP material maintains a grain size below 15 μm even at 1200°C. It should be noted that the 2Mo steel does not contain the Al and V grain-refining additions as do the 9Ni-4Co steels; therefore, these elements do not appear to be a factor in the grain-coarsening resistance of the RSP steels.

The time dependence of grain growth at high temperatures was examined in the 9Ni-4Co-0.2C steel. Results for the RSP material are shown in Fig. 3 and for the conventional material in Fig. 4. It is found that the RSP material maintains a grain size below 10 μm for up to 16 h. at 1100°C (this is about one-third of the grain size of the conventional material after 15 min. at 900°C). At longer times, however, a discontinuous coarsening reaction begins. The points indicated in parentheses represent the approximate average grain size from a pronounced duplex structure. This discontinuous coarsening begins at earlier times at higher temperatures. The abrupt change of grain size and formation of a duplex

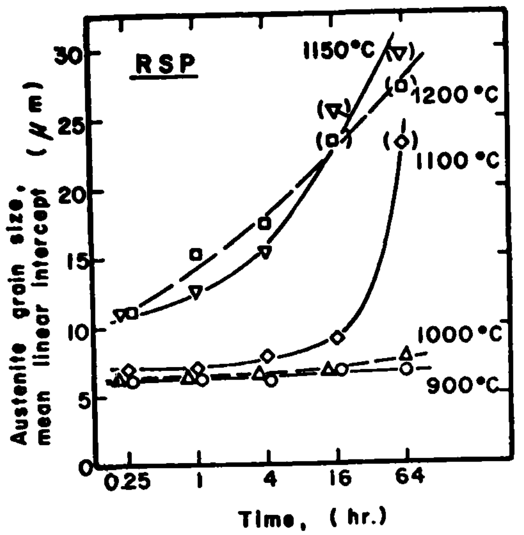

Fig. 3. Isothermal grain growth of rapidly solidified 9Ni-4Co-0.2C steel.

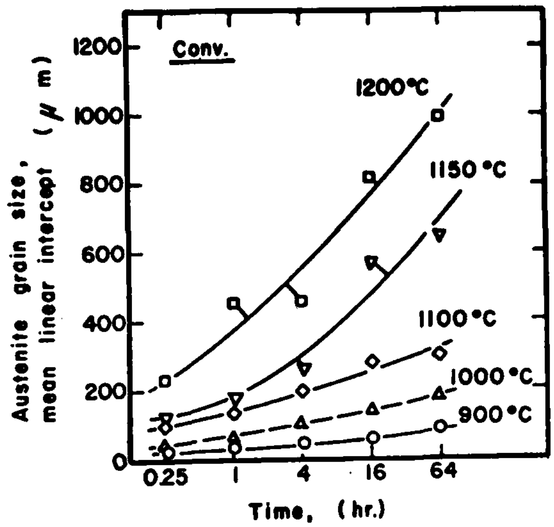

Fig. 4. Isothermal grain growth of conventionally processed 9Ni-4Co-0.2C steel.

structure are characteristic of the abnormal grain growth accompanying the break-away of boundaries from pinning particles. Over this same temperature region, the conventional material of Fig. 4 undergoes smooth (though rapid) coarsening with a fairly uniform grain-size distribution consistent with normal grain growth. These results are suggestive of the presence of boundary-pinning particles in the RSP material which begin to dissolve or coarsen in the range of 1100-1200°C, and which are not operative in the conventionally processed material.

While the behavior of the average grain size presented in Figs. 1-4 gives a good indication of the overall coarsening behavior of these materials, some peculiar aspects of the RSP material are best shown by the microstructures presented in Fig. 5. These represent longitudinal sections of the 9Ni-4Co-0.2C extruded RSP steel. Fig. 5a shows the as-extruded material. Superimposed on a fine background structure is an array of elongated domains ∿30 μm in width and ∿300 μm in length. As ellipsoidal cylinders, these domains would correspond in volume to a spherical particle of ∿65 μm diameter. Hence, these elongated domains are believed to represent individual prior powder particles. It was noted that this domain structure was not as well-defined in the center-most portion of the extruded bar, and this center region generally showed slightly faster grain-growth kinetics. These differences may be associated with non-uniform deformation during extrusion.

Grain growth during austenitizing of the RSP material appears to occur in three basic stages. At temperatures below ∿1000°C, there is a very slow growth of equiaxed grains within the prior particle domains. During this stage the prior particle boundaries gradually disappear as indicated in Fig. 5b. At higher temperatures and longer times, an unusual stage of nonuniform grain growth occurs. Although prior particle boundaries no longer etch, a "necklace" structure of enlarged grains develops, comprising elongated regions having the size and shape of the prior particle domains. Fig. 5c shows this structure at the same magnification as Figs. 5a and b, and Fig. 5d shows a lower magnification view which emphasizes the directionality of this microstructure. It appears that grains within some prior particle domains coarsen abruptly to a size limited by the lateral dimensions of the domains. Grain growth then proceeds by similar abrupt coarsening in other prior particle domains which "catch up" to the grain size of the first domains. At the end of this stage, a fairly uniform equiaxed grain structure is attained, Fig. 5e, and this then gradually coarsens.

The abrupt coarsening within prior particle domains might be related to the

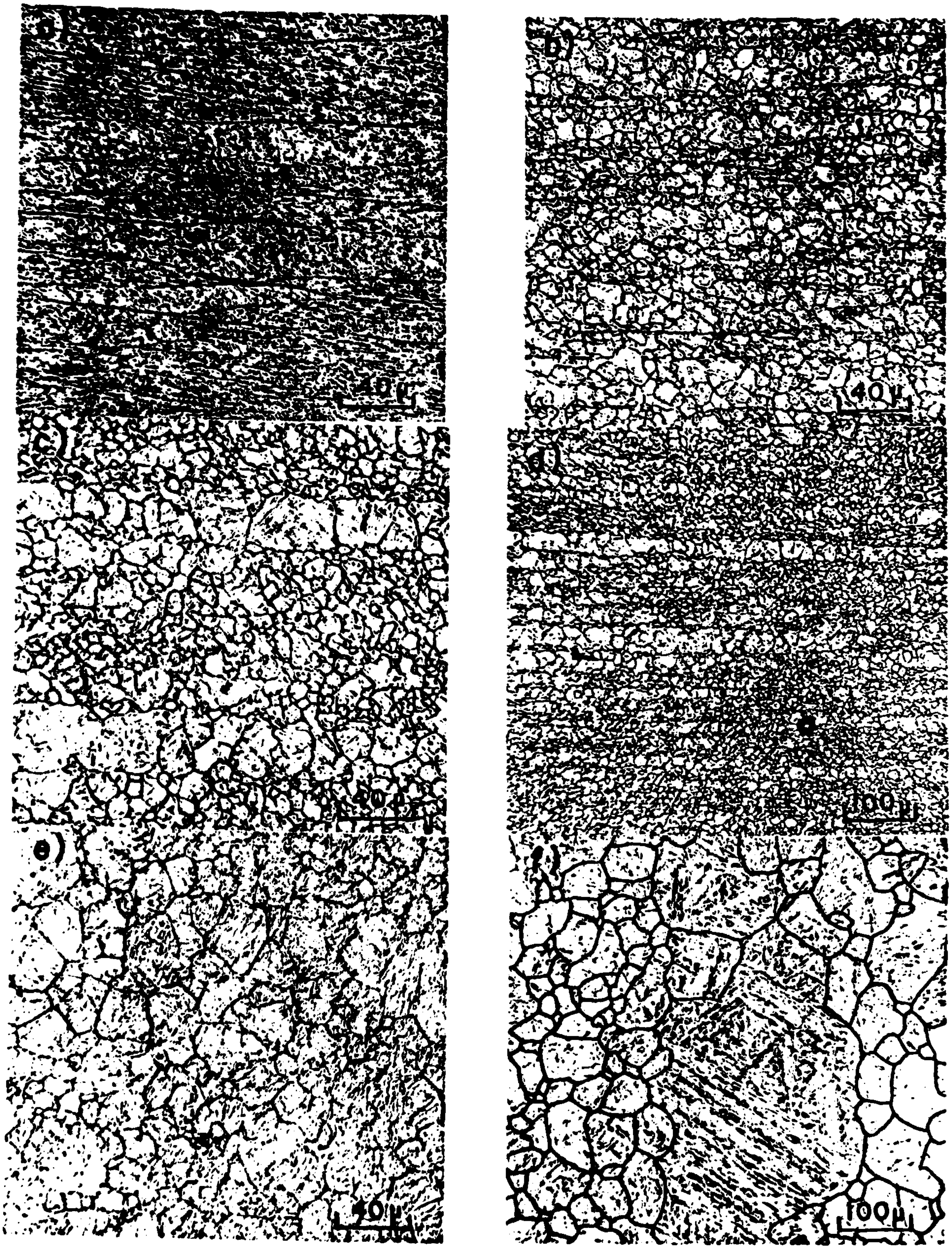

Fig. 5. Microstructure of rapidly solidified 9Ni-4Co-0.2C steel: a) as-forged, showing prior particle boundaries; b) austenitized 1 hr. at 900°C, showing elimination of prior particle boundaries and equiaxed grain structure; c & d) "necklace" structure developed by coarsening within prior particle domains after 15 min. at 1100°C; e) equiaxed structure developed after completion of coarsening within prior particle domains, 15 min. at 1200°C; f) discontinuous coarsening after 16 hr. at 1200°C. Boundaries in (f) have been retouched for clarity.

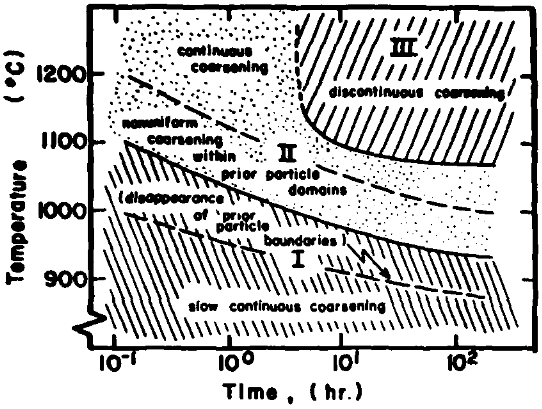

Fig. 6. Temperature-time-reaction plot for the three stages of grain coarsening in rapidly solidified 9Ni-4Co-0.2C steel.

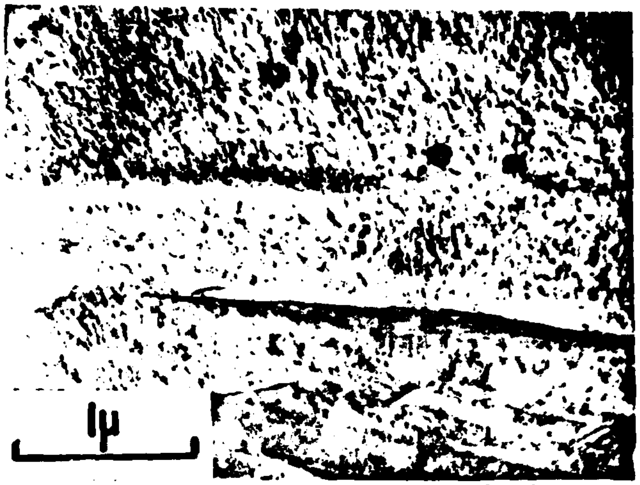

Fig. 7. Bright-field transmission electron micrograph of 2Mo steel austenitized at 1100°C.

boundary-particle breakaway process responsible for the rapid coarsening in the conventional material above 900°C, although the process in the RSP material is occurring in a temperature range ∿150°C higher. That different domains coarsen at different temperatures and times suggests that there may be important variations in the microstructure of different powder particles. It is also clear from this type of coarsening behavior that there is strong grain boundary pinning at prior particle boundaries, as evidenced in particular by some of the rectangular-shaped grains in Fig. 5c.

The third stage of grain growth in the RSP material is the discontinuous coarsening mentioned earlier. This is initiated by the appearance of a few abnormally large grains, as depicted in Fig. 5f, which grow to consume a fairly stable finer grain matrix. This discontinuous reaction is probably associated with a breakaway of grain boundaries from pinning particles at the prior powder-particle boundaries.

The temperature-time conditions for the various stages of coarsening just discussed are summarized in Fig. 6. It is particularly noteworthy that one-hour austenitizing treatments can be applied at temperatures up to 1200°C without the onset of the discontinuous coarsening reaction.

An indication of possible boundary-pinning particles responsible for the grain coarsening resistance of the RSP steels can be obtained from some preliminary electron microscopy. Fig. 7 shows a bright-field transmission electron micrograph of the RSP 2Mo steel austenitized 15 min. at 1100°C. The ∿0.1 μm particles apparent in the figure were commonly observed throughout foils of the RSP material, and were identified as MnS by X-ray fluorescence analysis in the STEM. In an earlier extraction replica study of alloy carbide precipitation in the 9Ni-4Co RSP steels (3) similar fine sulfides were observed and identified by STEM X-ray microanalysis as sulfides principally of Mn with substantial amounts of Fe and small amounts of V, Cr, and Ni. The occurrence of a fine dispersion of manganese sulfides is consistent with the observation in an RSP high-sulfur stainless steel that rapid solidification refines the average sulfide size by several orders of magnitude (4). Although the sulfides so far observed have not been preferentially located at grain boundaries, the conditions for discontinuous coarsening of the RSP steels roughly correlate with the expected temperature range for the onset of dissolution of MnS in commercial purity steels (5) as occurs in the phenomenon of "overheating". A higher density of sulfides can be expected at prior particle boundaries if surface segregation of sulfur occurs during solidification as suggested by observations in rapidly solidified nickel-base alloy powders (6). Further electron microscopy, particularly in the region of the prior particle boundaries, is planned to further assess the role of sulfides in the unusual

grain-coarsening resistance of RSP steels. That the coarsening resistance may be due to fine sulfides suggests the interesting possibility that microstructural constituents which may be otherwise inert (or even deleterious) can be made to play a beneficial role in microstructure through their fine dispersion via rapid solidification.

The high-temperature grain-coarsening resistance of RSP steels might be used to advantage in improving the fracture toughness of high strength steels. It is known that the sharp-crack fracture toughness (K_{IC}) of higher-carbon (≥.40C) ultrahigh-strength steels, such as 4340 and 300M, can be substantially enhanced by the use of high austenitizing temperatures up to 1200°C (7-9). This toughening effect can be attributed primarily to the dissolution of void-initiating inclusion particles (9, 10). However, even though K_{IC} is increased, the blunt-notch toughness (e.g. Charpy energy) and tensile ductility (reduction in area) are found to decrease due to the excessive grain coarsening which normally attends such high austenitizing treatments (10, 11). The coarsening resistance of RSP steels should allow the use of these high austenitizing treatments without the accompanying deleterious grain growth. Hence there is the prospect that K_{IC} can be improved without sacrificing Charpy energy and ductility, provided of course, that the stable boundary pinning particles are sufficiently finely dispersed that they themselves do not limit toughness. The elimination of coarse inclusions through rapid solidification and high austenitizing treatments may also be beneficial to fatigue strength.

In pursuing improved fracture properties via rapidly solidified powder processing, a note of caution is warranted concerning powder contamination. Preliminary tensile tests revealed that the ductility of the steels used in this investigation is limited by cross-contamination with nickel-base alloy powders. New material has now been obtained with a greatly improved level of cleanliness, and fracture toughness will soon be investigated. If substantial improvement of the toughness of high strength steels is to be achieved via RSP, it is evident that the utmost level of powder cleanliness will have to be maintained.

Conclusions

RSP steels show a remarkable resistance to high-temperature grain coarsening relative to conventionally processed steels, allowing the use of austenitizing temperatures as high as 1200°C without excessive grain growth. Strong grain-boundary pinning appears to be associated with prior powder-particle boundaries. Evidence so far suggests that the pinning may be due to fine sulfides. The use of high austenitizing treatments in RSP steels may allow substantial improvement of sharp-crack fracture toughness (K_{IC}) without the loss of Charpy energy and ductility associated with excessive grain growth.

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